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Atomic layer deposition instrument for in-situ environmental TEM imaging of ALD process

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Atomic layer deposition (ALD) is an important and relatively new thin-film deposition technique compared to PVD and CVD. ALD allows deposition of oxides, metals, semiconductors, polymers and layered combinations thereof. The possibility of depositing uniform, conformal films on various surfaces, including high-aspect-ratio structures, makes ALD a very important technique for nanofabrication [1].

The ALD process consists of a sequential inlet and purging of gas phase precursors and counter-reactants over a substrate surface. An important advantage of ALD is the precise thin-film thickness control, due to the self-limiting behavior of each deposition cycle. To expand further the use of ALD a deeper understanding of the deposition process is necessary. In particular comprehension of the nucleation and growth of the film during the early stages of deposition is very important for the fabrication of ultra-thin films.

Real-time visualization of ALD processes will allow in-depth and fundamental understanding of the deposition mechanisms. We propose imaging of ALD using an environmental transmission electron microscope (ETEM), which permits the study of gas-solid interactions by inlet of gases inside the microscope specimen chamber [2]. To perform *in-situ* ALD studies, it is necessary to obtain a stable and reproducible ALD deposition inside the ETEM. It is also important to make sure that ALD reactions do not contaminate or damage the microscope components, which may deteriorate the imaging capability of the ETEM. As an ALD model system, we selected Pt ALD using oxygen and a solid metalorganic Pt precursor. Pt ALD is selected because of its ETEM favorable *ALD window*.

Dedicated to the above study, we report on the design and development of a portable ALD system (Fig. 1 and 2). The system is a cold-wall reactor and it consists of three main parts: a reactor chamber, a gas injection system (GIS) with ALD valves for injection of gaseous precursors and a vacuum system for reactor and GIS pumping. The reactor chamber is built with features and parameters resembling the ETEM specimen chamber [3], i.e. reactor volume, pumping geometry, and sample holder heating system. Two different pump lines connected to the same scroll pump perform pumping of the chamber. One pump line (rough pumping) pumps the system from atmospheric pressure to deposition pressure (mbar range), while the other line (process pumping) pumps the chamber during ALD deposition through 200 μm apertures made by laser machining. The reactor pressure can be adjusted between $5 \cdot 10^{-3}$ mbar and atmospheric pressure by variation of the carrier gas flow. The reactor pressure during the ALD process is kept in the range 5-10 mbar, which is equivalent to the ETEM specimen chamber, and the pressure is also comparable with our commercial hot-wall ALD reactor (Picosun). Inlet and purging of precursors are performed using a 15 sccm continuous flow of nitrogen. The precursors are let in via ALD Swagelok valves with 10 ms response time. Fast valve switching time divides a precursor pulse of few seconds into several pulses of shorter duration, and hence avoids excessive precursor vapor pressure drop to obtain more uniform precursor adsorption. Gas flows rates are set using mass flow controllers (MFC) with flow range 0-200 sccm. The sample holder is an aluminum plate fixed by stainless steel posts (low heat conductivity) to the reactor chamber lid, which provide thermal insulation of the reactor walls from the sample holder. Sample temperature is feedback controlled using a vacuum compatible cartridge heater and a thermocouple passing through the reactor lid. The low Pt metalorganic precursor vapor pressure can be increased by a custom precursor bottle holder with a temperature control system.

Once we have thoroughly tested our ALD system, we can connect it to the ETEM as precursor inlet unit for *in-situ* imaging of ALD process (Fig. 3).

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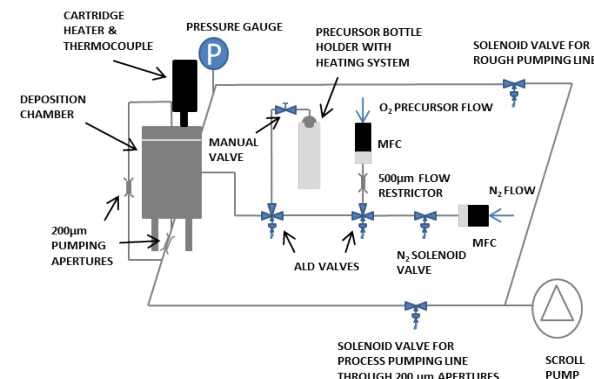


Figure 1. Schematic drawing of the portable deposition system.

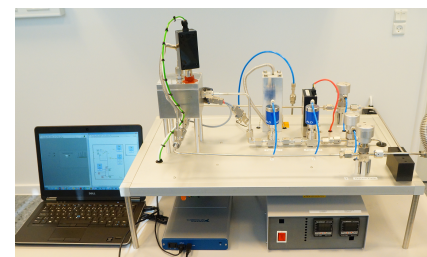


Figure 2. Picture of the portable ALD system with DAQ device and temperature controllers.

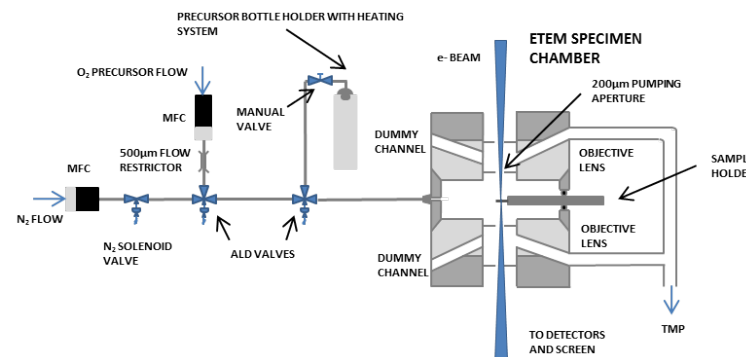


Figure 3. Connecting the portable ALD system and differentially pumped ETEM specimen chamber.